Quality and shelf life of orange juice aseptically packaged in PET bottles

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Abstract

A packaging study of orange juice aseptically packaged in bottles using different materials and filling procedures was conducted to determine their influence on the evolution of juice quality and shelf life. Glass, multilayer PET (polyethylene terephthalate) and monolayer PET bottles were used. A characterisation study including DSC thermograms, intrinsic viscosity and scanning electron microscopy (SEM) photographs let identify the different material used, taking part in multi- and monolayer PET bottles for juices packaging. Monolayer PET showed the lowest retention of ascorbic acid during storage and shelf life compared with multilayer PET and glass. But if different factors, such as oxygen scavenger, liquid nitrogen drop addition in headspace during filling, aluminium foil seal in screw-cap and refrigeration temperatures, are combined with the monolayer PET bottles, orange juice shelf life can be extended and values similar to glass and multilayer PET bottles can be obtained.

Keywords: Orange juice; Aseptic packaging; PET bottling; Glass bottling; Shelf life

1. Introduction

Over the past few years the consumption of fresh citrus fruits has decreased in developed countries. Processed products, especially juice, have become more popular because they are easier to be consumed and are products of a high nutritional quality.

Among citrus juices, orange juice is the most appreciated and consumed because of its pleasant taste and its high content of vitamin C. Quality and shelf life determination of an orange juice is strongly based on vitamin C evolution during storage although there are other quality parameters such as colour and flavor characteristics that are also very important (Lee & Coates, 1999; Meydev, Saguy, & Kopelman, 1977; Zerdin, Rooney, & Vermuë, 2003).

Vitamin C is an essential nutrient for humans and, because of its high antioxidant power it provides protection against the presence of free radicals participating in the prevention of many diseases (Tannenbaum, Archer, & Young, 1985).

However, and because of its nature, vitamin C is oxidized and lost during the storage period of the juice. The rate of degradation of the vitamin C highly depends on the storage conditions (Kabasakalis, Siopidou, & Moshatou, 2000). Among the factors affecting vitamin C loss in packed orange juice, temperature, dissolved oxygen and oxygen barrier provided by the container material should be considered (Tannenbaum et al., 1985).

The choice of the packaging material for fruit juices is a crucial point regarding shelf life and much research has been done on this subject (Askar, 1999; Ebbesen, 1998; Siegmud, Derler, & Pfannhauser, 2004; Zerdin et al., 2003). However, aspects relating to product quality during the shelf life of the product as well as costs should be considered, too.

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Because of its excellent mechanical properties, its clearness, UV resistance and good oxygen barrier properties, PET (polyethylene terephthalate) is increasingly used in food packaging. Liquid foods such as milk or oil are some of the products for which PET has been proposed as a packaging material (Moyssiadi et al., 2004; Tawfik & Huyghebaert, 1998; Zygoura et al., 2004). As for orange juice bottling, PET is being actually used eventhough limited research work has been published on the matter (Muratore et al., 2005). In fact, when considering a packaging material for orange juice bottling it would be appropriate to select the material that better matches quality objectives, shelf life, storage temperature and costs of the product even though in most cases juice manufacturers do not exactly know particular behaviour of the commercial PET packaging materials for bottling.

As mentioned before, the presence of oxygen is one of the main factors responsible for orange juice deterioration (Soares & Hotchkiss, 1999). The adverse effects of dissolved oxygen on fruit juice quality have been investigated by many researchers and include degradation of ascorbic acid, increased browning and growth of aerobic bacteria and moulds (Eiroa, Junqueira, & Schmidt, 1999; Kennedy, Rivera, Lloyd, Warner, & Jumel, 1992; Meydev et al., 1977; Soares & Hotchkiss, 1999; Solomon, Svanberg, & Sahlstrom, 1995). Traditional methods for juice packaging aim to reduce the exposure of the juice to oxygen through the use of high barrier materials such glass or foil laminates in brickpacks, with or without nitrogen flushing (Zerdin et al., 2003) or improving gas barrier of PET by blending with aromatic polyamides (Hu et al., 2005). The use of oxygen scavengers with an appropriate packaging material can further reduce the presence of dissolved oxygen in the juice or present initially in the headspace. This technique has been investigated for the packaging of solid foods (Baiano & Del Nobile, 2005; Martínez, Djenane, Cilla, Beltrán, & Roncalés, 2006; Muratore et al., 2005; Sanchez Silva, López Hernández, & Paseiro Losada, 2005) and orange juice in plastic bags (Zerdin et al., 2003).

Although materials with a low permeability to oxygen as glass or multilayer PET, have been proposed by many author as the best to preserve nutritive and sensory quality of liquid foods (Siegmud et al., 2004; Sobek, 2003) the cost of these packaging materials is relatively high. The use of lower priced materials, as monolayer PET with a higher permeability to oxygen, in conjunction with techniques aimed at reducing the presence of oxygen inside the package, appears as an attractive alternative for oxygen sensitive liquid product as orange juice.

The aim of this study was to characterize different PET packaging materials used for orange juice bottling and to determine the effect of different filling procedures and storage temperatures on the quality and shelf life of orange juice packed in monolayer PET bottles.

2. Material and methods

2.1. Packaging materials characterization

In this study, bottles made of different packaging materials were used. The studied bottles were (a) a 1 L clear glass bottle, (b) a 1 L clear three layers multilayer PET bottle, 380–410 μm in thickness, (c) a 1 L clear monolayer PET bottle, 550–600 μm in thickness and (d) a 1 L clear monolayer PET bottle monolayer PET with oxygen scavenger (OS), 450–500 μm in thickness. PET bottles were supplied by Tecnopet, S.A. (Spain). The bottles were blown from preforms using a commercial blow molding machine (SBO Universal, SIDEL).

The thermal analysis of samples of the different material layers from above bottles (multilayer PET, monolayer PET and monolayer PET with OS) were carried out using a Mettler Toledo model DSC822e. The weights of samples were in the range from 4 to 10 mg. In order to delete the previous thermal history, the samples were initially heated to 300 °C, kept at this temperature for 5 min and, then, rapidly cooled to room temperature. After this thermal treatment, the samples were analysed from 30 to 300 °C at 10 °C/min (Pawlak, Pluta, Morawiec, Galeski, & Pracella, 2000). Two replicates were made for each experiment.

Analysis of intrinsic viscosity were carried out for each material layer sample in the cases of multilayer PET, monolayer PET and monolayer PET with OS using a Ubbelohde viscosimeter. Measurements were carried out at 25 °C and the dissolvent used was a mixture of phenol/1,1,2,2-tetrachloroethane (60/40, w/w). Intrinsic viscosity was calculated according to ISO 1628-1 standard for viscosity number dependence on concentration of polymer solution (Duarte, Paula e Silva, Branco, & Lins, 2004).

Thickness and structure of films for monolayer PET and monolayer PET with OS was analysed using scanning electron microscopy (SEM) with a Hitachi model S-3500N SEM.

2.2. Packaging and storage of orange juice

The orange juice (1 L per bottle) was transferred manually into the bottles of monolayer PET and monolayer PET with scavenger using a laminar air-flow cabin (Telstar bio-II-A model, BIOIIA) and in aseptic industrial filler into the bottles of glass and multilayer PET (at Agrumexport, S.A. facilities). Orange juice was manually bottled at 25 °C under nitrogen flushing in different PET monolayer bottles with or without aluminium-foil seal in the screw-cap (AL) and with or without a liquid nitrogen (LN) drop addition in headspace resulting in the following treatments (for monolayer PET bottles):

Treatment 1: PET + oxygen scavenger, with aluminium-foil seal in the screw-cap.

Treatment 2: PET + oxygen scavenger, without aluminium-foil seal in the screw-cap.

Treatment 3: PET + oxygen scavenger, with aluminium-foil seal in the screw-cap and LN addition.
Treatment 4: PET + oxygen scavenger, without aluminium-foil seal in the screw-cap and LN addition.
Treatment 5: PET with aluminium-foil seal in the screw-cap without LN addition.
Treatment 6: PET without aluminium-foil seal in the screw-cap and without LN addition.
Treatment 7: PET with aluminium-foil seal in the screw-cap and LN addition.
Treatment 8: PET without aluminium-foil seal in the screw-cap and LN addition.

Twenty bottles were prepared for every treatment and one bottle was analysed every 15 days.

Dimethyl dicarbonate, a sterilising agent, was only added to the orange juice manually packed in monolayer PET bottles at a concentration of 250 mg/L of juice. The use of this sterilising agent avoids microbial growth and because of its fast decomposition into natural constituents of juice neither the nutritional value, the taste, nor the colour of the juice is affected.

The vitamin C concentration of the juice was determined at different storage time.

Samples were stored at 4 °C and at 25 °C in darkness.

The shelf life estimation was determined according to the Industry of Juices and Nectars from Fruits and Vegetables of the European Union that establishes a limit of 20 mg/100 mL orange juice for ascorbic acid at shelf life expiration date (Polydera, Stoforos, & Taoukis, 2003).

2.3. Determination of ascorbic acid

The ascorbic acid (AA) content was determined by a HPLC analytical procedure using a Waters Alliance 2695 (separation module), Waters 2487 dual absorbance detector, Waters Atlantis column dC 18.5 μm, 4.6 × 150 mm, flow of 1.4 mL/min, trifluoroacetic acid (0.01%) in water as mobile phase and the wavelength of 260 nm as operational conditions.

The orange juice was centrifuged at 4500 rpm and 4 °C for 15 min. Supernatant was filtered in GiroVent 0.45 μm and placed in the HPLC device for chromatography analysis. The obtained values were calculated as mg/L of AA. Each sample was prepared and analysed in triplicate.

2.4. Microbiological testing

A microbiological investigation of the juice packed under the above-mentioned conditions, was carried out on the same samples as those for ascorbic acid, to be sure that microorganisms did not interfere with the oxidation of vitamin-C by competitively consuming the available oxygen.

Acidophilic microorganisms (lactic acid bacteria and yeasts) were determined because they have been shown to be the major microbial contaminants of citrus juices (Alwazeer, Delbeau, Divies, & Cachon, 2003). All the samples were analysed for duplicate.

2.5. Colour measurement

Juice colour was measured using a CR-200 Minolta Chroma meter (Minolta, Chuo-Ku, Osaka, Japan) with an 8 mm measuring area. A Minolta standard-white reflector plate was used to standardise the instrument under CIE (Commission International de L'Eclairage) illuminating C conditions. Samples of orange juice were filled into 60 mL glass assay tubes and CIELab values were determined. All samples were analysed in triplicate (Alwazeer et al., 2003; Esteve, Frígola, Rodrigo, & Rodrigo, 2005).

The L^*, a^*, b^* colour values were determined using the 1976 CIELAB system.

The colour parameters in all treatments were measured as a function of storage time at 25 and 4 °C using for this purpose Eqs. (1) and (2).

$$\mathrm{Cr} = (a^2 + b^2)1/2 \tag{1}$$

where: Cr: chroma, is the grade of quantitative difference of Hue parameter with reference to grey colour, a is a measure of red tones and it varies from $-a$ to $+a$ ($-a$ = green, $+a$ = red), and b is a measure of yellow tones and it varies from $-b$ to $+b$ ($-b$ = blue, $+b$ = yellow).

$$^{\circ}H = \mathrm{Arc}\,tg(b/a) \tag{2}$$

where: $^{\circ}H$: Hue angle, is the qualitative attribute of colour and it defines the difference of a colour with referent to grey, L represents the brightness measure and the luminosity at range from 0 to 100 (100 = white; 0 = black).

2.6. Dissolved oxygen in orange juice

The dissolved oxygen content of the juice was measured using a probe of dissolved oxygen (oxygen probe type FYA640O2 by ALMEMO-Systeme). The bottles were opened and the oxygen probe was immediately inserted into the juice for 5 min (Zerdin et al., 2003).

2.7. Sensory analysis

For the sensory evaluation an informal panel of 12 untrained assessors evaluated the degree of acceptability of samples based on taste, acidity, sweetness, aroma, colour and general impression. A five-point hedonic scale was used for all characteristics and a mean value of 3 was chosen as the limit of acceptability for shelf life determination.

2.8. Statistical analysis

Results were submitted to analysis of variance (ANOVA) with significance defined as $P < 0.05$ using the Statgraphics statistical software.

3. Results and discussion

3.1. Packaging materials characterization

Figs. 1 and 2 show typical DSC thermograms obtained for multilayer PET and monolayer PET samples, respectively.

Glass transition temperatures and melting points are shown in Table 1, for each material.

From DSC thermograms it is shown that two materials take part in multilayer PET, these were identified as polyethylene terephthalate (PET) and the aromatic polyamide Nylon MXD6 poly(*m*-xylylene adipamide) (Hu et al., 2005).

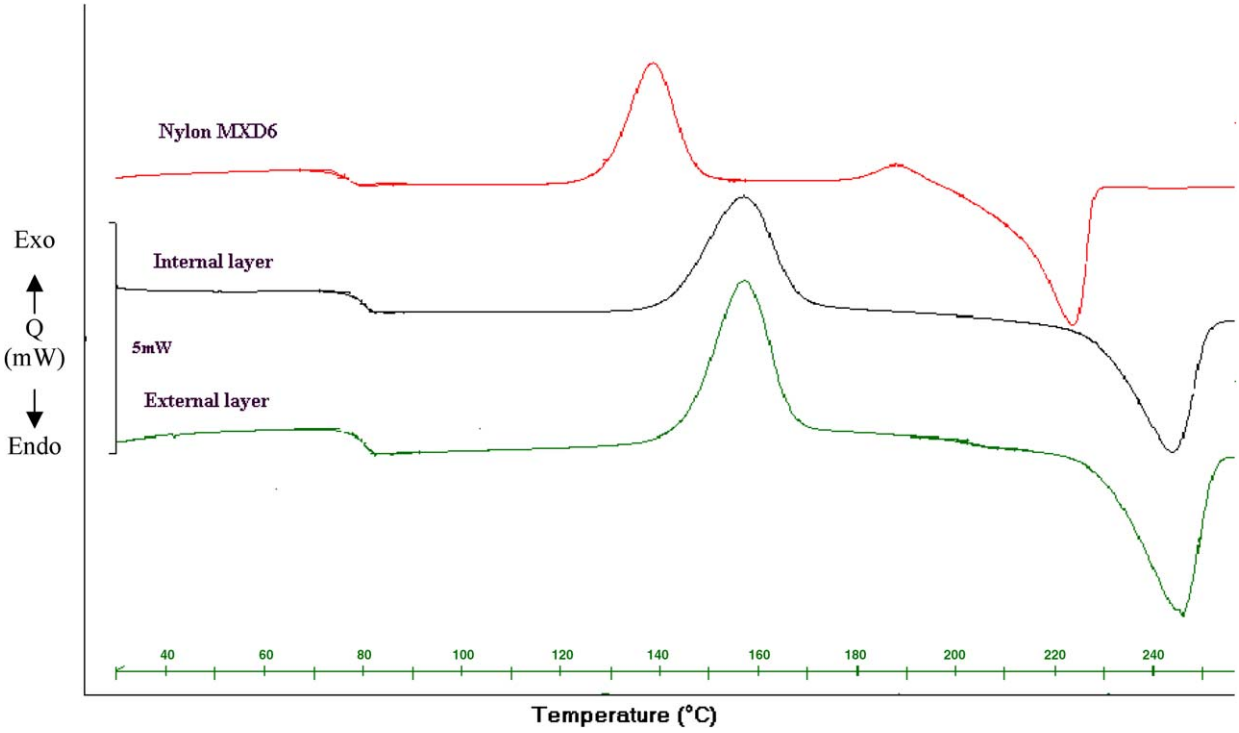

Fig. 1. DSC thermogram for multilayer PET (with Nylon MXD6 interior layer).

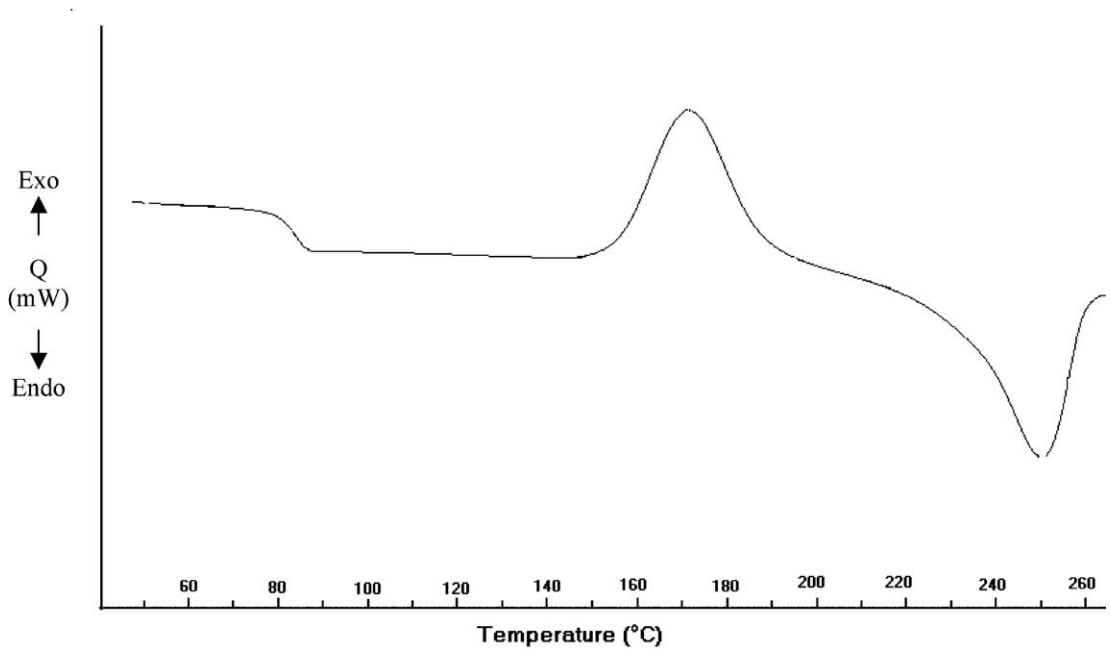

Fig. 2. DSC thermogram for monolayer PET.

Table 1
Average values and standard deviation of glass transition temperatures and melting points for different PET materials

Material	Glass transition temperature (°C)	Melting point (°C)
Multilayer PET (with MXD6)	81.14 (2.30)	247.78 (4.46)
Monolayer PET	82.82 (0.70)	250.57 (0.76)
Monolayer PET with OS	82.25 (0.80)	250.27 (1.04)

On the other hand, it was observed that the addition of the oxygen scavenger to monolayer PET did not change thermal properties of the material because both parameters, glass transition temperature and melting point, remained basically the same.

Intrinsic viscosity values obtained were 0.715 (0.628–0.761) dL/g, 0.744 (0.714–0.808) dL/g and 0.788 (0.731–0.790) dL/g for multilayer PET, monolayer PET and monolayer PET with OS, respectively. These values are of the same order that those found in the literature for different classes of PET (Bikiaris & Karayannidis, 1996; Brooks & Giles, 2002; Hu et al., 2005; Pawlak et al., 2000).

SEM micrographs enables distinguish easily three layers in multilayer PET (Fig. 3a). These layers have been identified as two outside layers of PET and an interior thin layer of aromatic polyamide Nylon MXD6 poly(*m*-xylylene adipamide). For thickness determination of individual layers, the more compact areas were selected. Thickness of multilayer PET was 396 μm being the thickness of every individual layer 196, 47, and 153 μm. Thickness of monolayer PET and monolayer PET with OS were 575 and 463 μm, respectively (Fig. 3).

3.2. Acid ascorbic content evolution

Figs. 4 and 5 show the content of ascorbic acid of orange juice packed in monolayer PET without oxygen scavenger, multilayer PET and glass bottles and stored at 4 and 25 °C, respectively.

Bottles stored at 4 °C were found to better preserve ascorbic acid in orange juice than those stored at 25 °C. This result agrees with other authors as Ayhan, Yeom, Zhang, and Min (2001), Esteve et al. (2005), Kabasakalis et al. (2000), and Zerdin et al. (2003).

The material which presented the best retention of ascorbic acid was glass followed by multilayer PET. The simple monolayer PET bottle presented a poor retention of ascorbic acid. Being oxygen one of the main factors that contributes to ascorbic acid degradation and considering that headspace was the same for all bottles the only factor that can explain these differences in ascorbic acid retention is oxygen permeability. Results indicate that glass was the material that presents the lowest oxygen permeability followed by multilayer and monolayer PET. This is in agree-

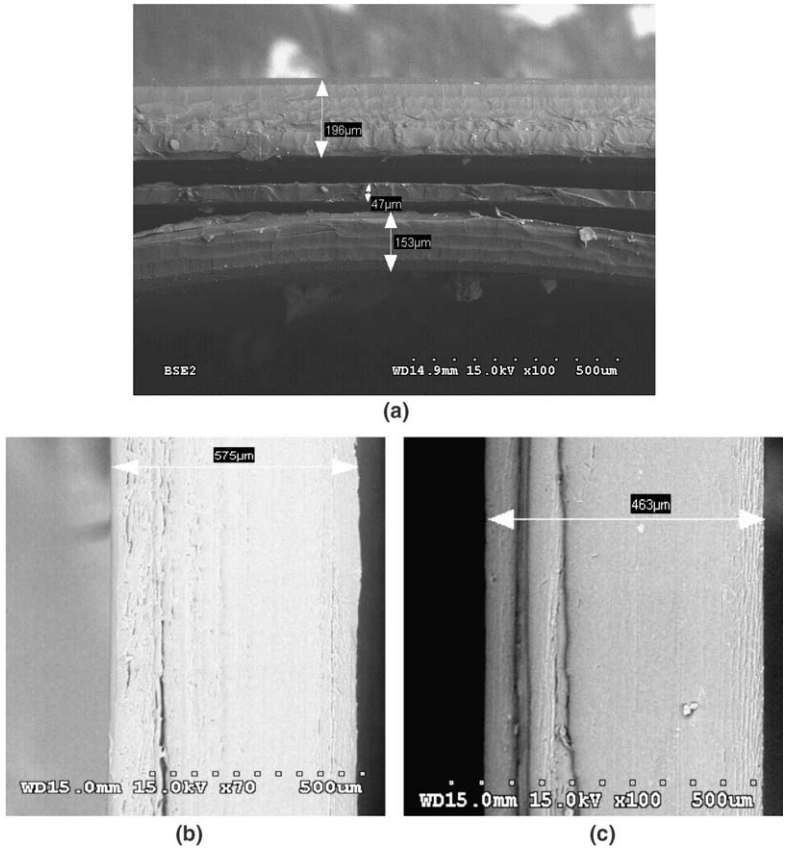

Fig. 3. SEM photographs of (a) multilayer PET, (b) monolayer PET and (c) monolayer PET with OS.

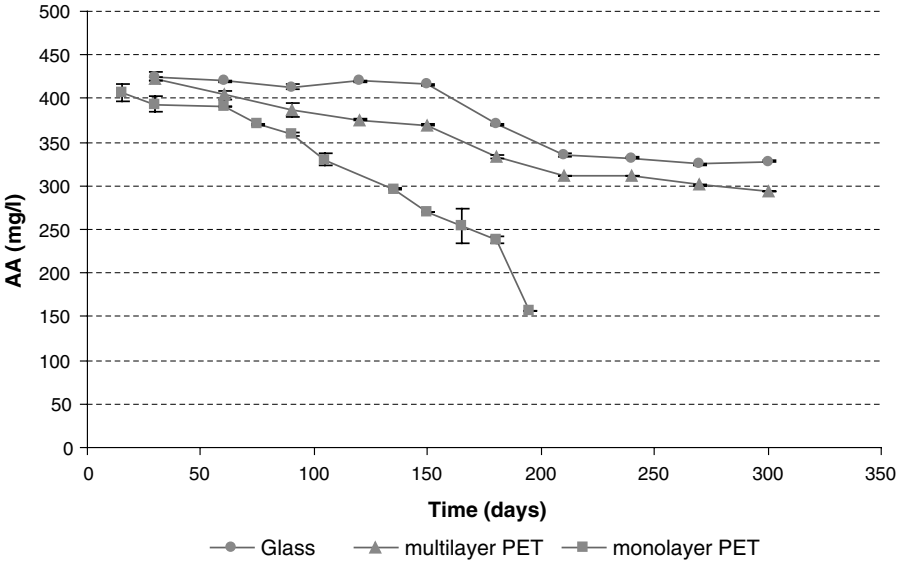

Fig. 4. Content of ascorbic acid in orange juice from PET and glass bottles stored at 4 °C.

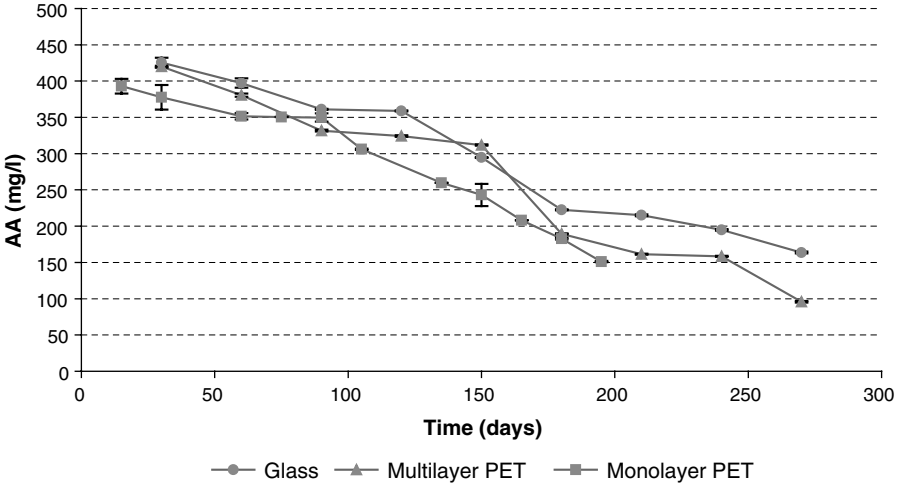

Fig. 5. Content of ascorbic acid in orange juice from PET and glass bottles stored at 25 °C.

ment with other works (Ayhan et al., 2001; Muratore et al., 2005; Sobek, 2003).

Considering the limit of 200 mg/L for shelf life estimation (Polydera et al., 2003), glass and multilayer PET bottles can reach more that 300 days at 4 °C and 250 days at 25 °C while, for simple monolayer PET, shelf life was around 180 days at 4 °C and a maximum of 160 days at 25 °C.

At the end of the studied period acid ascorbic retention was significantly higher at 4 °C than at 25 °C ($P < 0.05$).

3.3. Dissolved oxygen

Table 2 shows dissolved oxygen values at the end of the studied period (180 days) for the different monolayer PET bottling treatments. It is observed that for monolayer

Table 2
Dissolved oxygen content (average values and standard deviation) in orange juice stored under different treatments at day 180, using monolayer PET bottles

Treatment		O_2 (mg/L)	
		25 °C	4 °C
OS	LN without AL	0.0 (0.00)	0.2 (0.00)
	LN with AL	0.1 (0.05)	0.2 (0.00)
	AL without LN	0.2 (0.05)	0.2 (0.06)
	Without LN–AL	0.4 (0.1)	0.2 (0.01)
Without OS	LN without AL	0.4 (0.00)	0.4 (0.00)
	LN with AL	0.4 (0.05)	0.3 (0.03)
	AL without LN	0.4 (0.06)	0.4 (0.05)
	Without LN–AL	0.4 (0.05)	0.4 (0.05)

OS = oxygen scavenger; LN = liquid nitrogen; AL = aluminium-foil seal in the screw-cap.

Table 3
Colour parameters (average values and standard deviation) for orange juice stored at 4 and 25 °C and different monolayer PET bottling treatments

Time (days)	Parameter	Treatment			
		PET + OS 4 °C	PET 4 °C	PET + OS 25 °C	PET 25 °C
15	L^*	45.07 (0.01)	44.98 (0.33)	44.45 (0.04)	44.88 (0.06)
	$°H$	107.7 (0.02)	107.95 (0.27)	108.07 (0.33)	107.54 (0.66)
	Cr	18.55 (0.06)	18.04 (0.25)	17.92 (0.17)	18.58 (0.34)
60	L^*	44.93 (0.44)	43.91 (0.22)	44.08 (0.18)	43.54 (0.43)
	$°H$	107.49 (0.06)	107.33 (0.10)	107.9 (0.03)	107.19 (0.20)
	Cr	18.57 (0.04)	19.34 (0.51)	17.34 (0.10)	19.55 (0.18)
105	L^*	43.84 (0.35)	44.69 (0.04)	42.29 (0.06)	43.85 (0.35)
	$°H$	107.33 (0.04)	107.52 (0.09)	106.64 (0.11)	106.07 (0.04)
	Cr	18.92 (0.21)	18.19 (0.02)	18.9 (0.48)	17.39 (0.21)
180	L^*	41.67 (0.25)	41.36 (0.22)	41.57 (0.21)	41.44 (0.04)
	$°H$	102.62 (0.02)	102.6 (0.10)	102.34 (0.17)	100.83 (0.04)
	Cr	20.76 (0.24)	20.58 (0.23)	19.62 (0.23)	18.56 (0.08)

OS = oxygen scavenger.

PET with OS and using LN with aluminium seal caps, dissolved oxygen in juice was lower (at 4 and 25 °C). Similar results were found by Zerdin et al. (2003). In the absence of OS the factors aluminium foil seal and liquid nitrogen in headspace did not affect the amount of dissolved oxygen in product.

3.4. Colour evolution

Table 3 shows the evolution of colour parameters for orange juice packed in monolayer PET bottles with and without OS and stored at 4 and 25 °C. It is observed that colour parameters did not present significant variations until 60 days of storage for both temperatures, 4 and 25 °C. After this time, values of L^* and $°H$ start decreasing which indicates that juice is losing its brightness and light and slightly changes its colour from initial yellow to reddish tones. This change may be attributed to non-enzymatic browning. However, Cr values showed an increasing trend, meaning that colours defined by $°H$ are more intense.

Statistical analysis showed that, at the end of the studied period, only temperature had a significant effect ($P < 0.05$) on orange colour being the bottles stored at 25 °C the ones that experimented significant changes in colour.

3.5. Microbiological analysis

No microbial growth was detected in either of the bottles. This indicates a good manufacturing practice in pasteurization and aseptic filling of orange juice. These results indicate that spoilage of microorganisms was not a factor that interfered in shelf life determination of orange juice.

3.6. Shelf life study

Considering the limit for shelf life estimation of juice 200 mg/L of ascorbic acid, shelf life was determined for

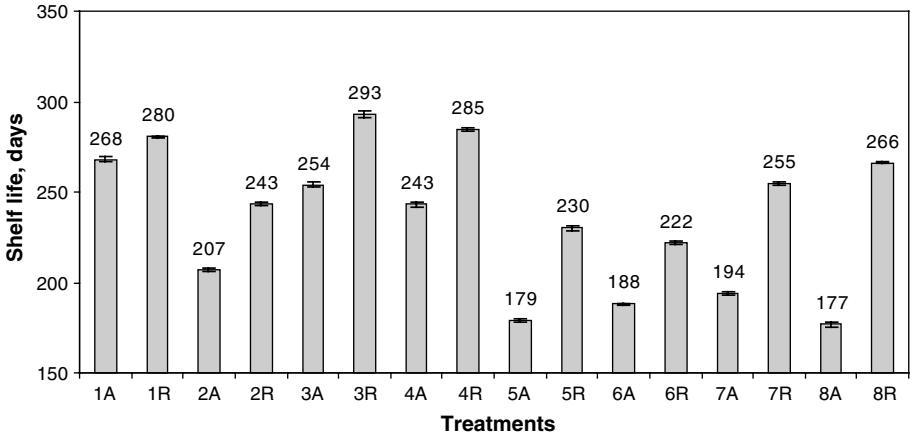

Fig. 6. Influence of different factors on shelf life of orange juice packaged in monolayer PET bottles. OS = oxygen scavenger, AL = aluminium-foil seal in the screw-cap, LN = liquid nitrogen, R = bottles stored at 4 °C, A = bottles stored at 25 °C. Number from 1 to 8 are the different treatments being 1: OS + AL; 2: OS; 3: OS + AL + LN; 4: OS + LN; 5: AL; 6: only monolayer PET; 7: AL + LN; 8: LN.

each monolayer PET bottling treatment (Fig. 6). Taking into account mean values from all treatments it is observed that bottles stored at 4 °C provide a longer shelf life (259 day) than those stored at 25 °C (213 days). This result indicates the interest of maintaining juice monolayer PET bottles juice at refrigeration temperatures.

Bottles packaged in monolayer PET with OS reached a longer mean shelf life than those without OS, 258 days versus 213 days. Finally bottles packaged with liquid nitrogen drop in headspace presented slightly longer shelf life (245 days) than bottles without it (226 days).

The longest shelf life was obtained combining the effects of OS, liquid nitrogen addition and aluminium foil seal. For this treatment, identified as 3, a shelf life up to 293 days was reached for juice stored at 4 °C (treatment 3R in Fig. 6). In all OS treatments the juice shelf life is longer than that actually demanded in the market for orange juice packaged in PET bottles, established as 180 days at 25 °C.

From the sensory point of view, all samples were evaluated as acceptable by the untrained panel and no significant differences between samples were found in any of the studied sensory characteristics.

4. Conclusions

Orange juice aseptically packaged in monolayer PET bottles presented a poor retention or ascorbic acid and shelf life was shorter than juice bottled in glass or multilayer PET. However, the PET bottling factors considered in this study had an additive effect on ascorbic acid retention and can extend juice shelf life to that provided by glass and multilayer PET. If orange juice is packaged in monolayer PET bottles containing an oxygen scavenger, with the addiction of liquid nitrogen in headspace and an aluminium foil seal in the screw-cap shelf life may exceed nine months at 4 °C and nearly eight months at 25 °C. Both values are much higher than those actually demanded on the market for juice aseptically packed in glass bottles at 25 °C for which a shelf life at 180 days has been established.

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